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U.S. Department  
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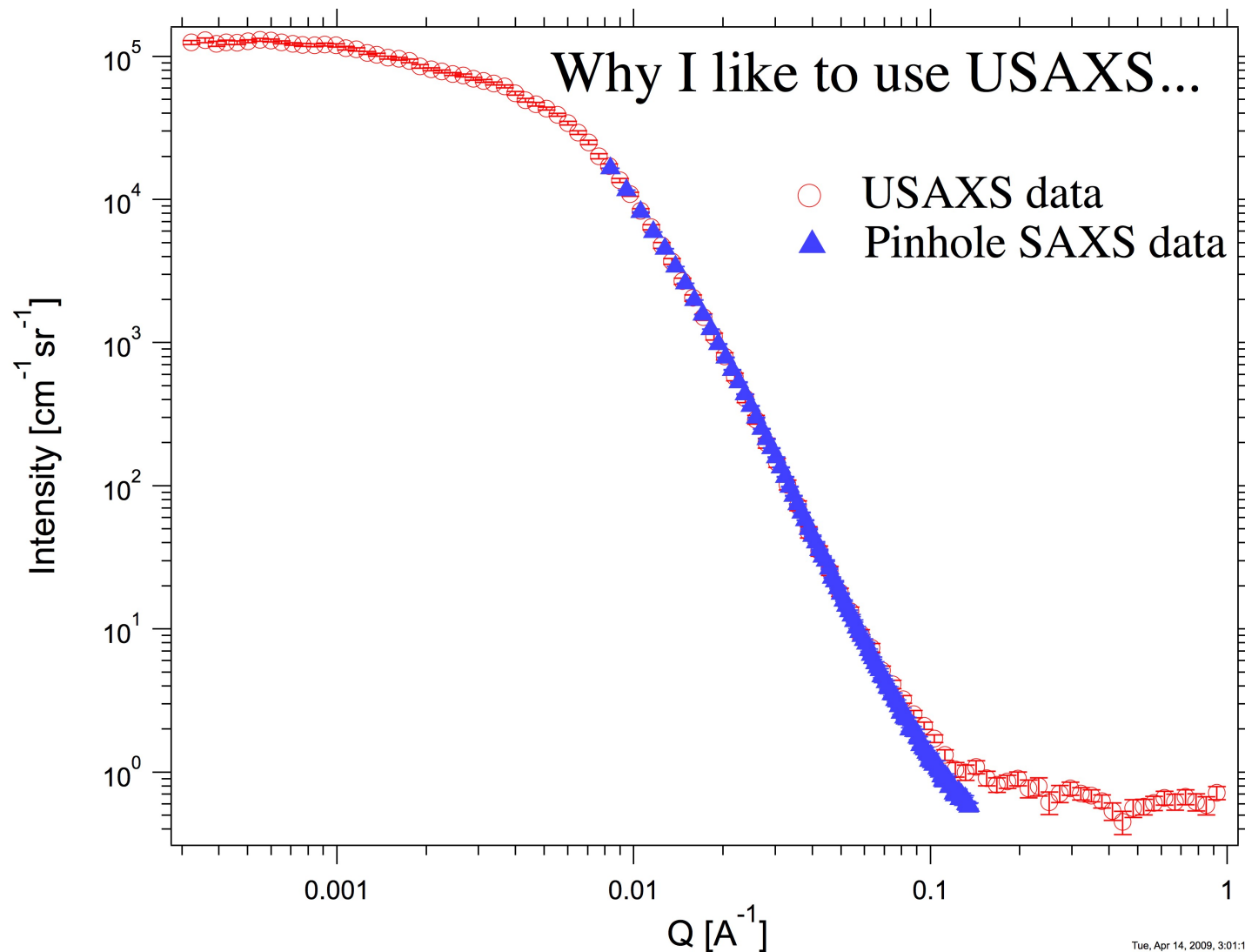
A U.S. Department of Energy laboratory  
managed by The University of Chicago

***USAXS instrument –  
instrumentation  
capabilities  
examples of science***

*Jan Ilavsky*

*Advanced Photon Source, Argonne National  
Laboratory*

*But first... Why do we need extended range instruments?*

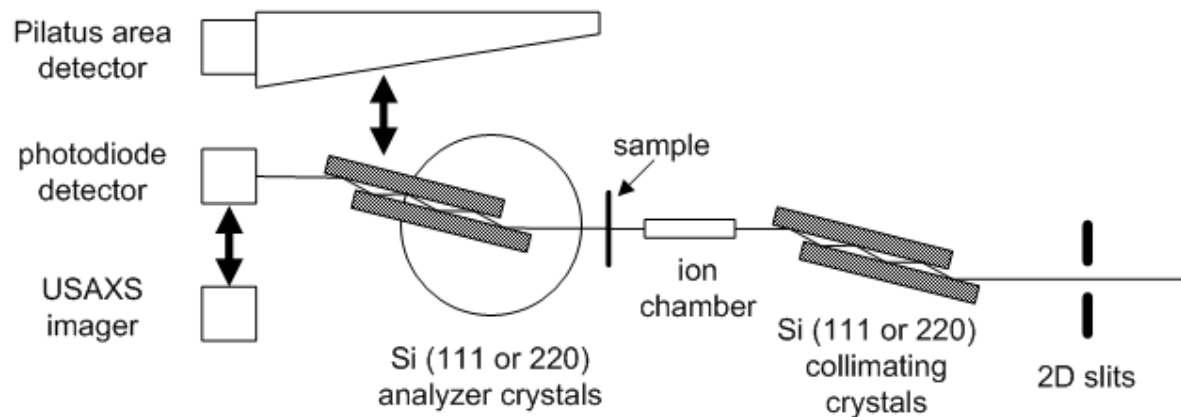


# USAXS/pinSAXS/USAXS-XPCS/WAXS

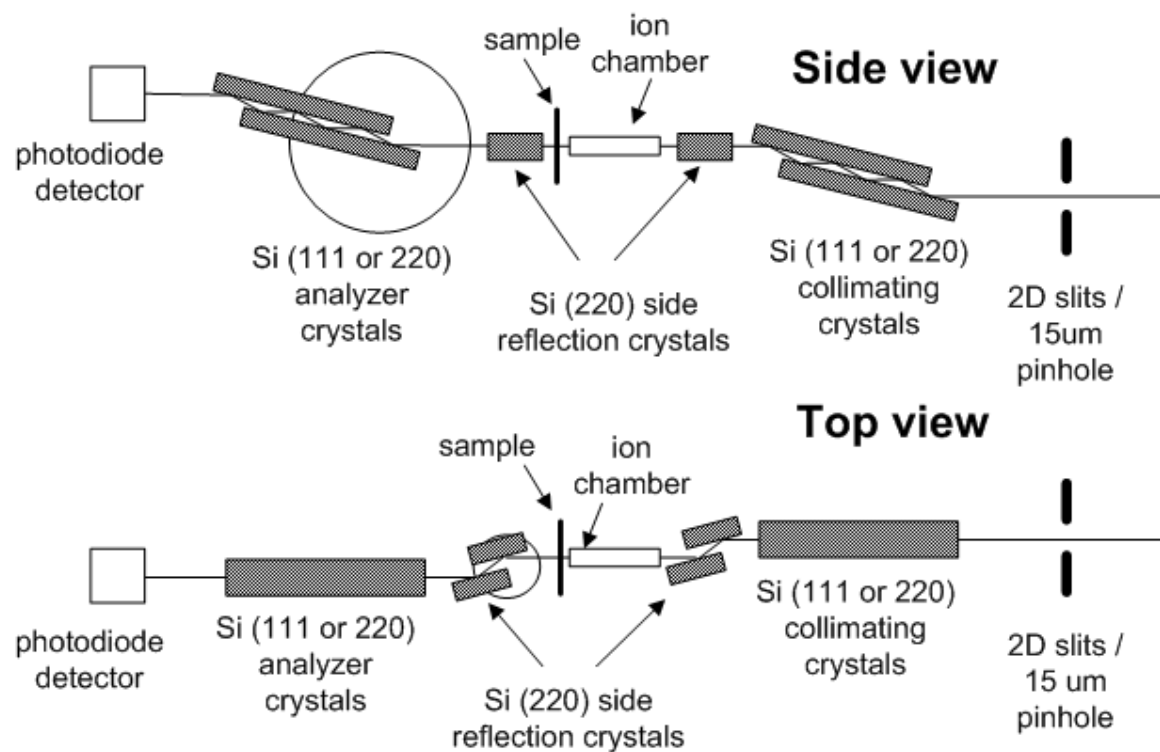
## Current parameters

- Range of X-ray energies 7 to 18 keV & 24keV (HE ops)
- 4 decades of Q range : 0.0001 to 1.0  $\text{\AA}^{-1}$  (HE 0.00002 to 1.0  $\text{\AA}^{-1}$ )
- Dynamic intensity range of over 9 decades
- Flexible beam size:
  - 0.02 – 1 mm vertical
  - 0.02 – 2 mm horizontal
  - 200  $\mu\text{m}^2$  minimum reasonable size
- 1-D and 2-D collimation geometries
- Combined Bonse-Hart & pinhole SAXS (“pinSAXS”) instrument
- WAXS optionally available (0.5 to 5.0  $\text{\AA}^{-1}$ )
- USAXS-XPCS geometry
- Standard-less absolute intensity calibration
- Imaging structures at various Q values (\*)
- User-friendly data reduction and evaluation software

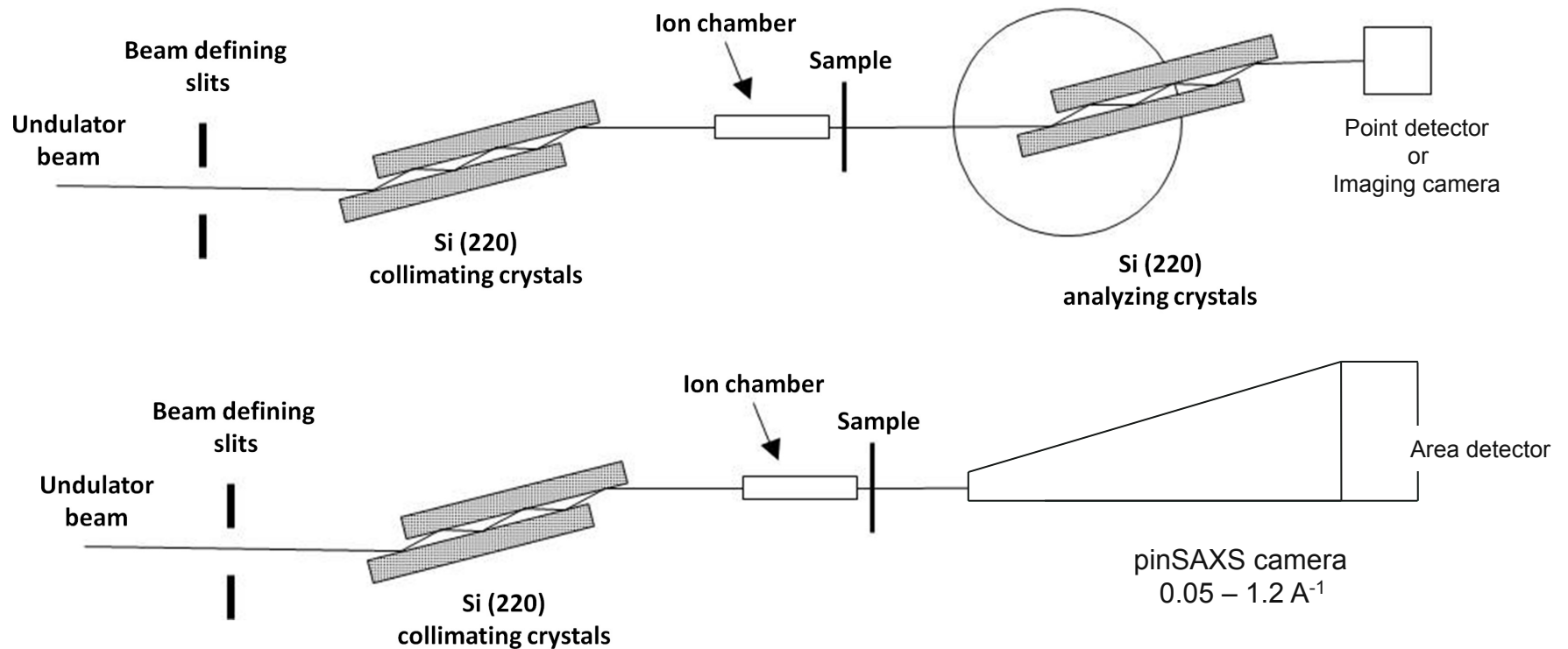
## 1D-Collimated USAXS w/optional SAXS



## 2D-Collimated USAXS/USAXS-XPCS



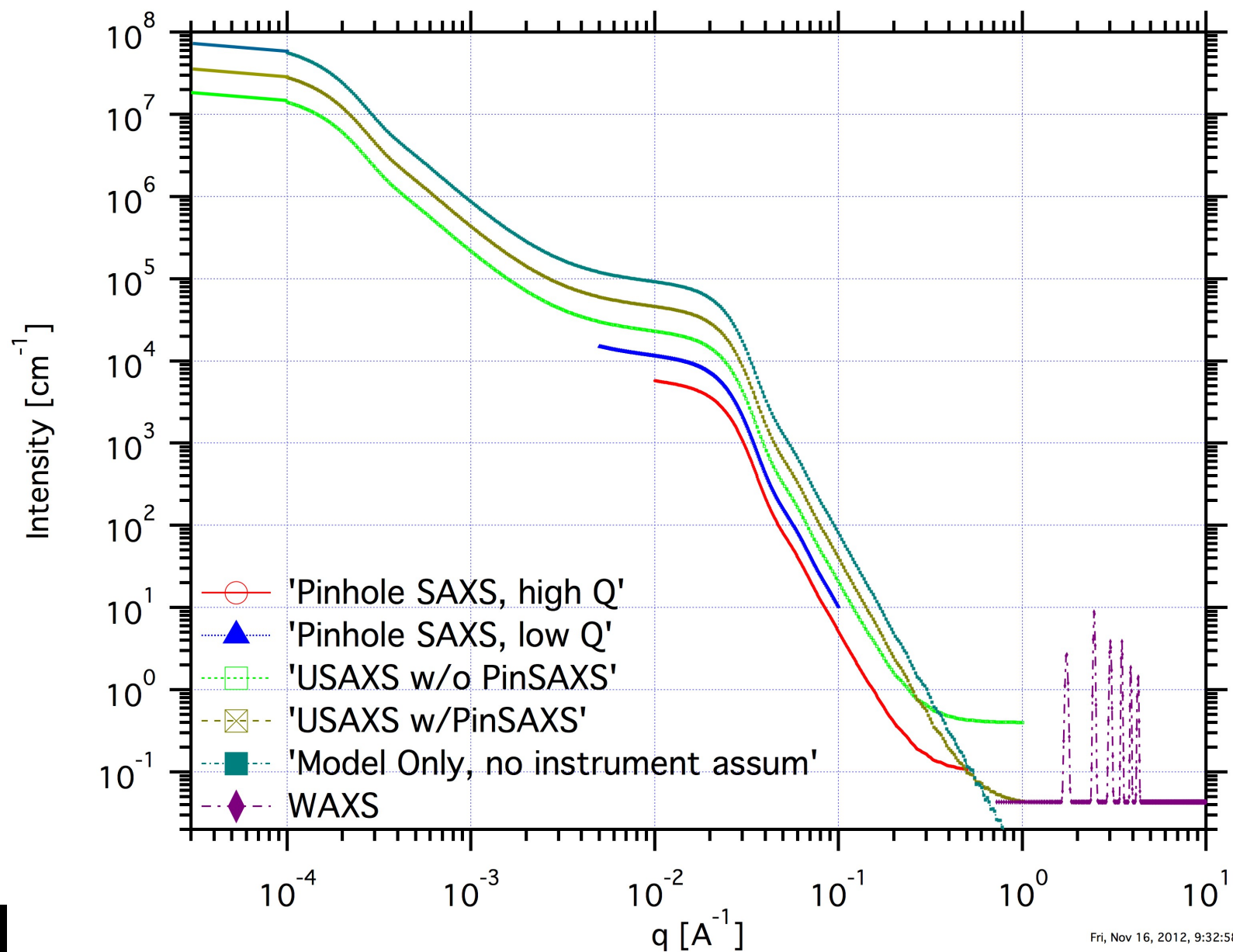
## 1D Bonse-Hart design USAXS, pinSAXS, & Imaging



### Measurements :

Intensity vs  $Q$  (slit smeared) combined USAXS & pinSAXS  
USAXS-Imaging (imaging materials at various  $q$  vectors)

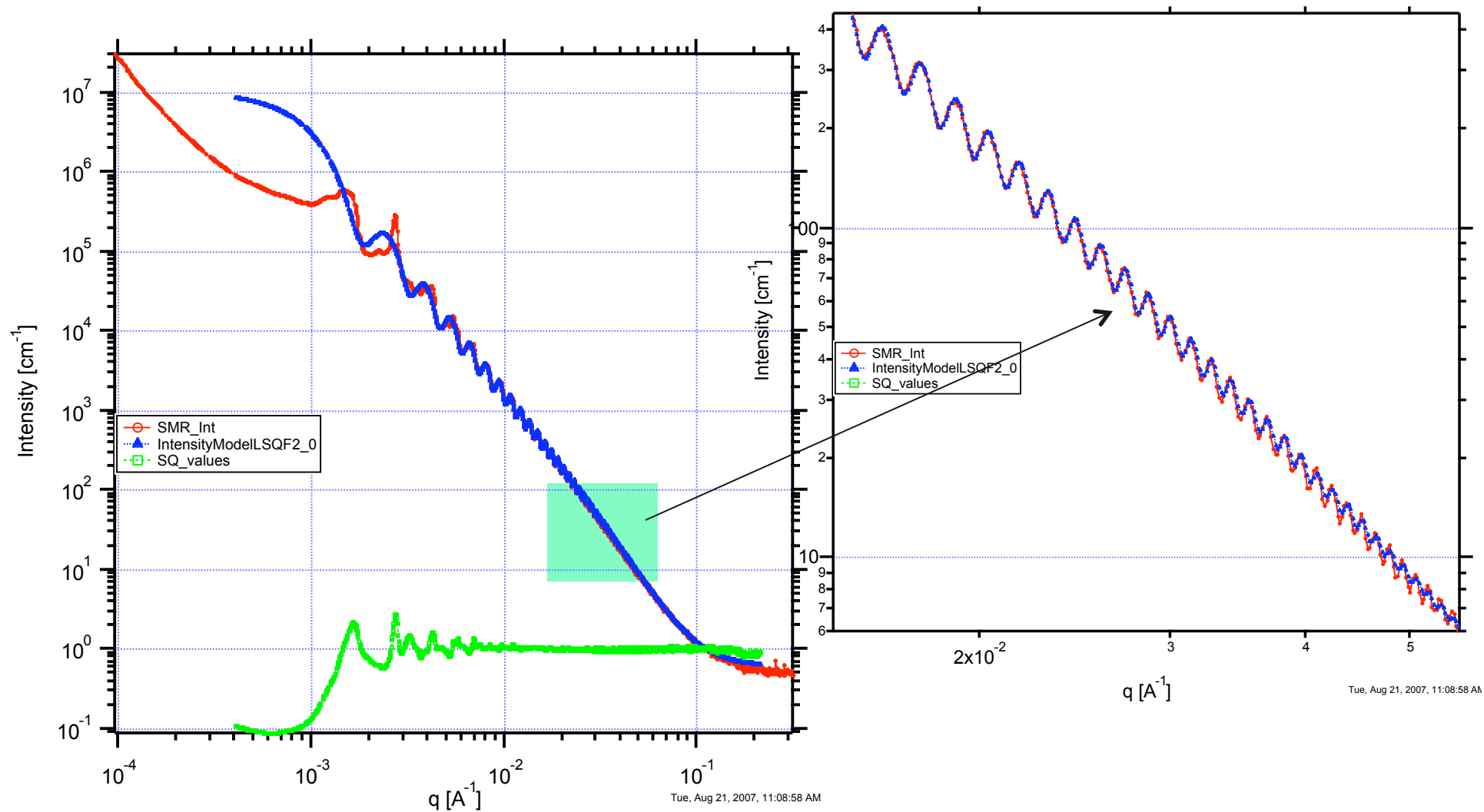
## Modeled HE-USAXS/WAXS capabilities



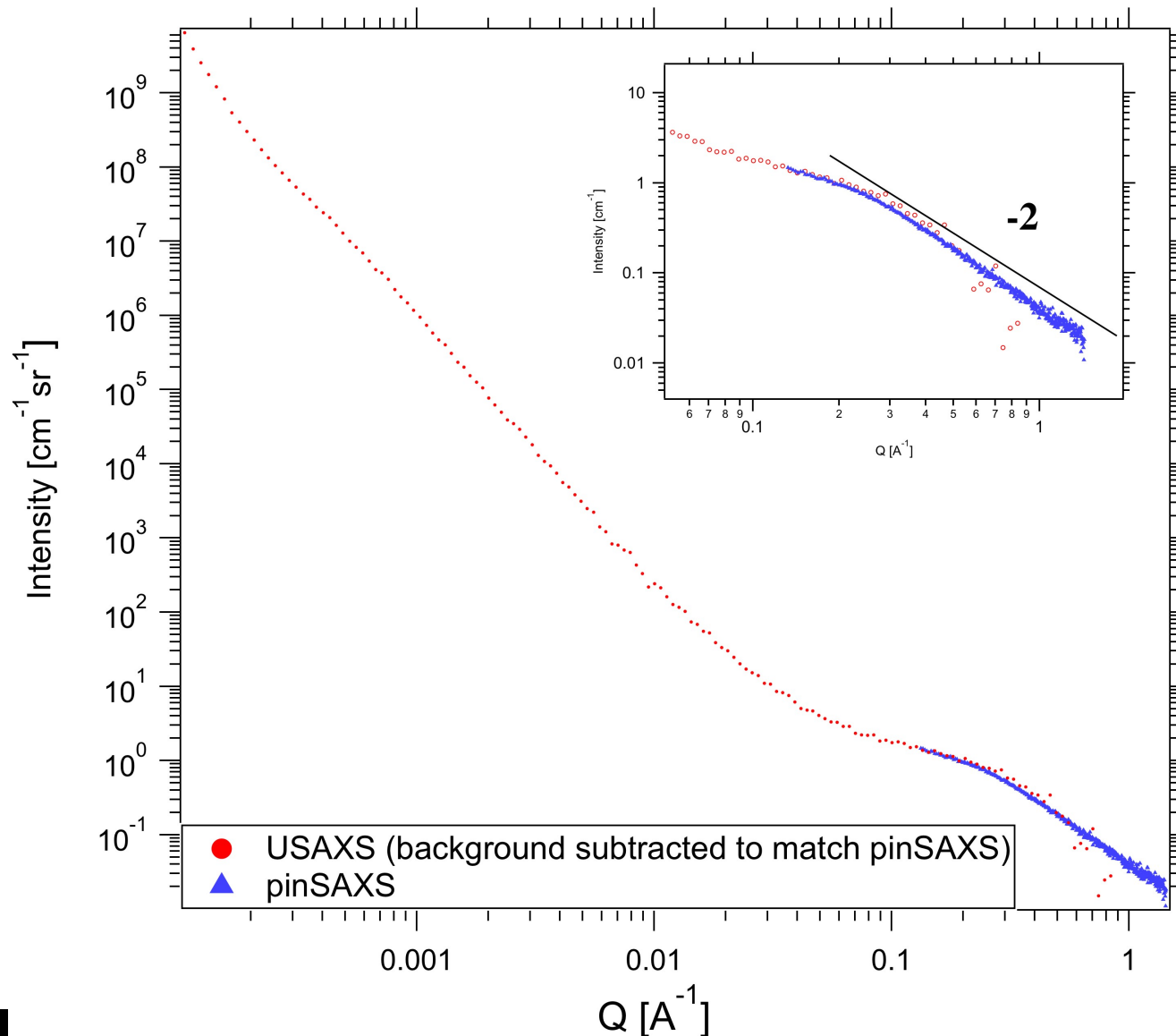
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# High q resolution

## Real World materials – mono sized distribution of spheres, powder



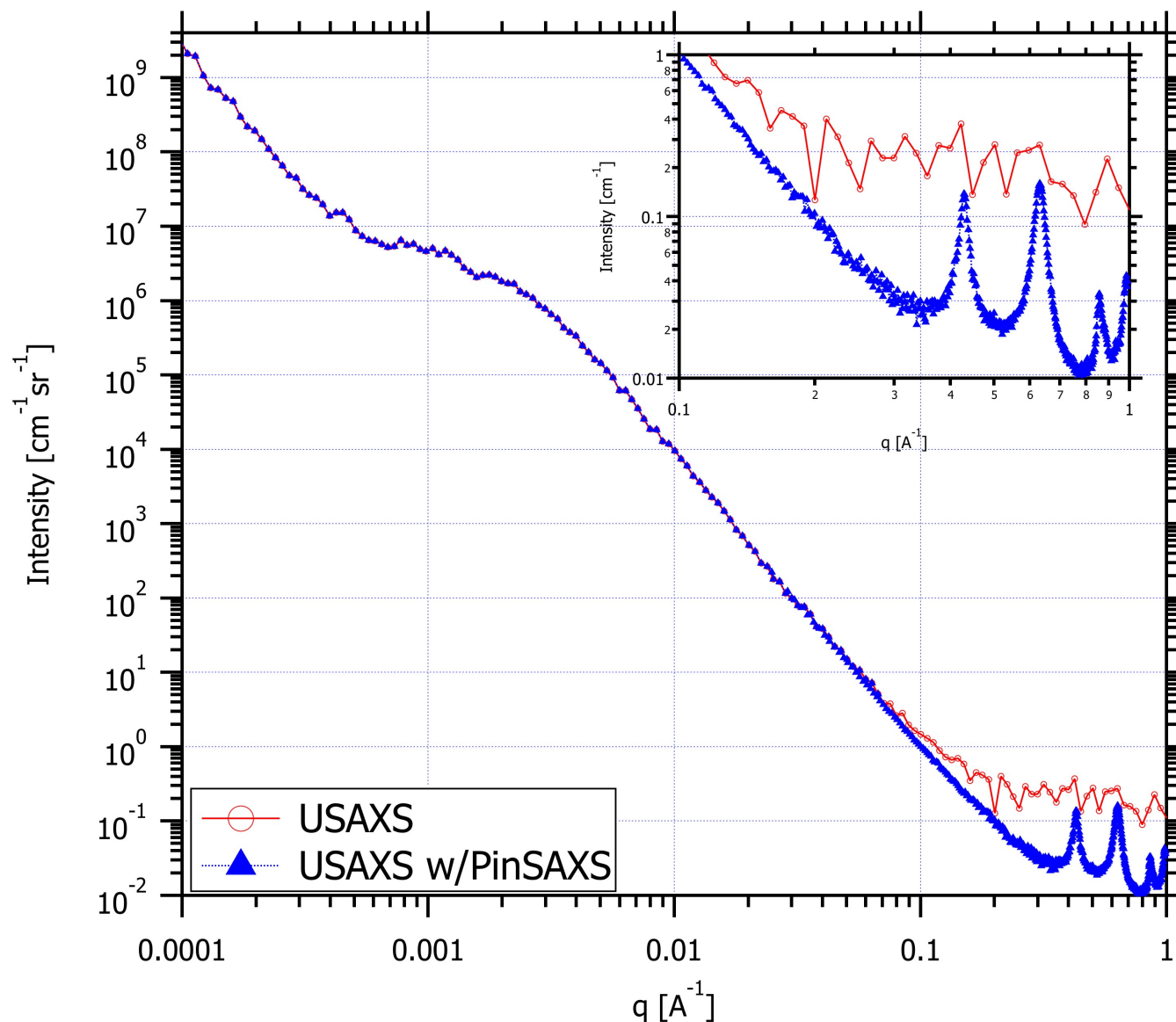
## Need for pinSAXS : Nanostructured carbon - data



- Peter Pfeifer's group (U Missouri) – studies of carbon materials for on-board gas storage for vehicles.
- Documents need for improved high  $q$  range

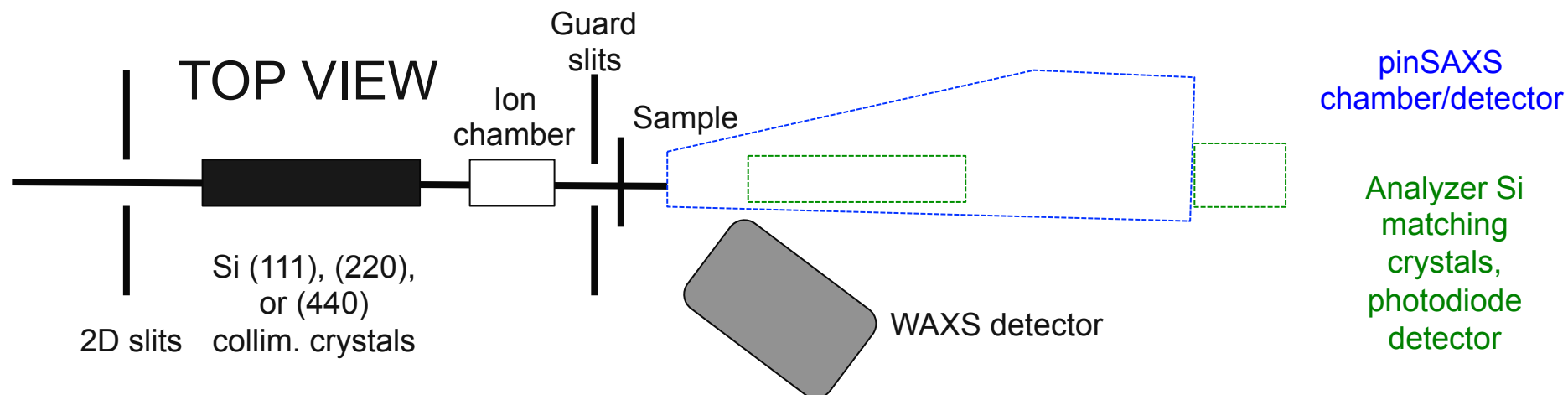


## Need for pinSAXS : Powder sample with small-angle diffraction



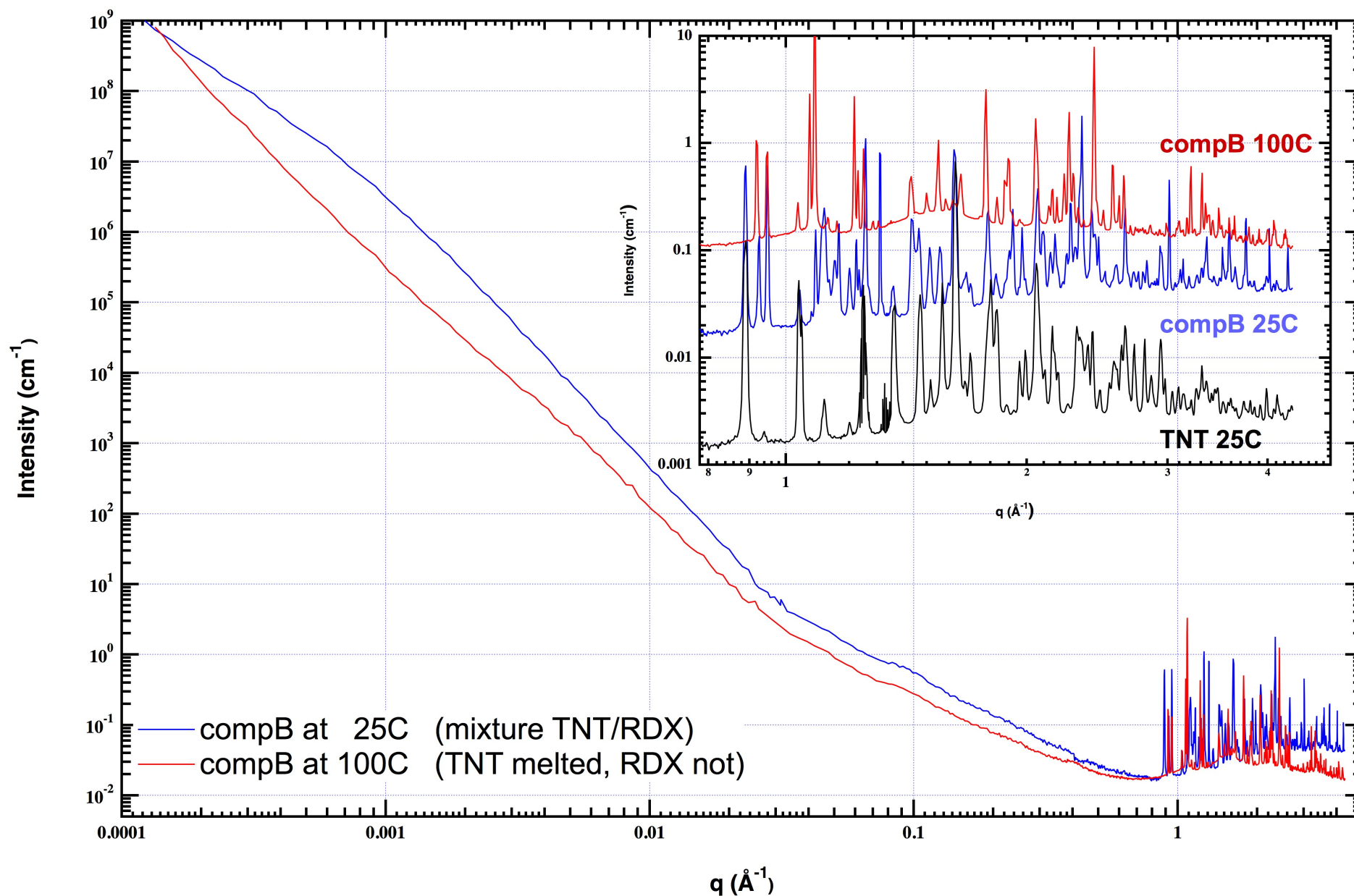
- Andrew Allen, powder sample with large structures
- Total dynamic range:  
> 11 decades
- Can see :
  - Powder particle size
  - Internal powder structure

# ***CAN WE MEASURE REAL WAXS? FOLLOW PHASE TRANSFORMATIONS FOR IN-SITU EXPERIMENTS?***



- Job: Add ability to measure 10 – 40 degrees angles with suitable area detector
- Conditions:
  - Do not impact USAXS and pinSAXS capability...
  - Make easy and transparent for users
- Solution: Use Pilatus 100k, 300k or similar detector (Pilatus 100k from APS equipment pool)
- Resolution tunable - given by size of detector, it's pixel size, beam size, etc.
- Tested in December 2012, user operations in 2013. Major success with users.
- Planned upgrade by procurement of Pilatus 300k-w to APS DP.

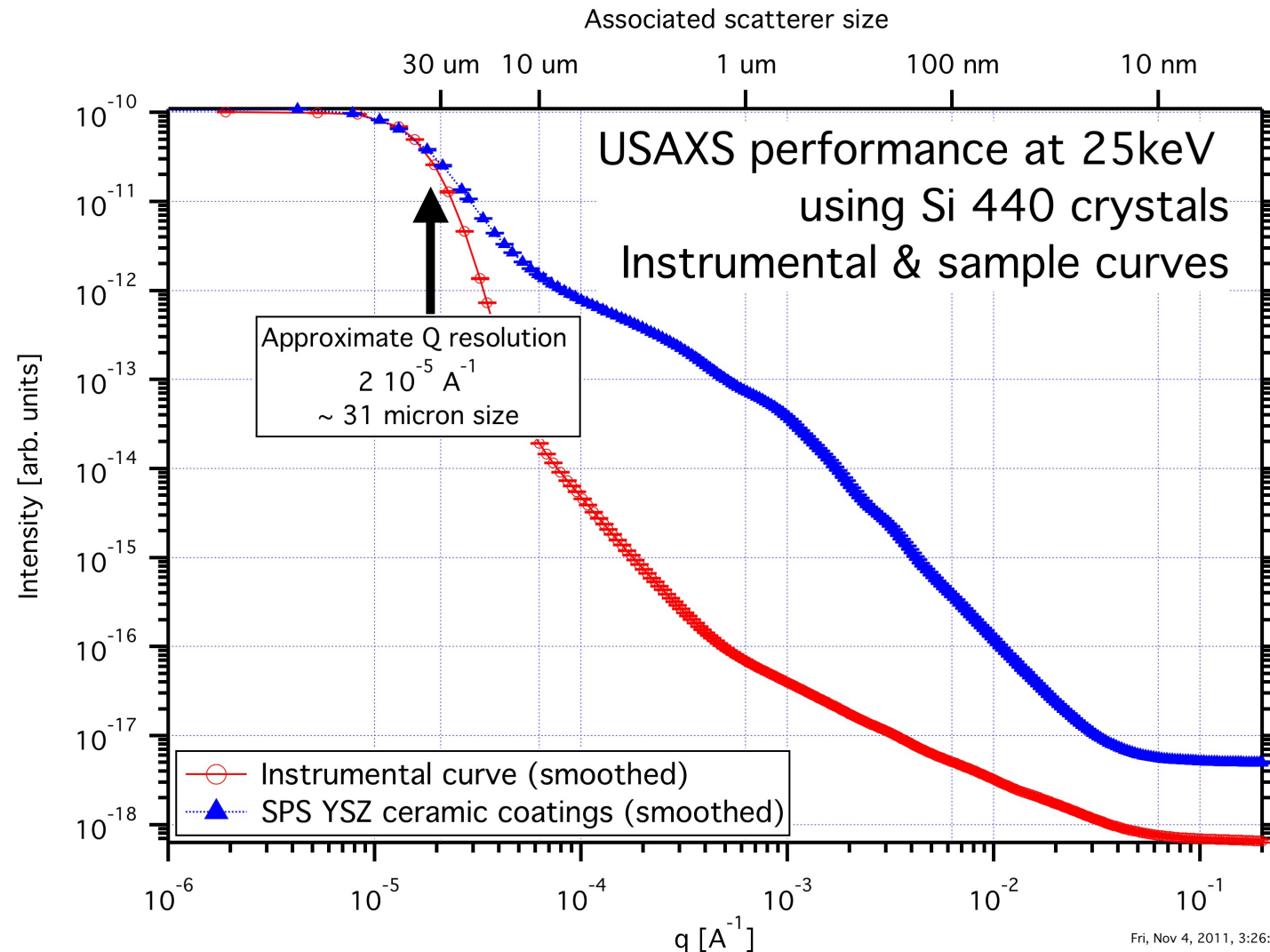
## Low-res (17keV) USAXS / pinSAXS / WAXS



## High energy operations

- Increase USAXS operational energy
  - Penetration through environments (heaters, reactors)
  - Thicker (more relevant) sample thicknesses
  - Higher-Z materials (Pu, but also cements, Pt, Au, ...)
  - Reduce multiple scattering (cement, porous ceramics)
- Using current Si (220) crystals as Si (440)
  - Easy change over (just change angle)
  - No additional expenses
  - No immediate commitment
  - Limits energy range :
    - $Si\ 220 = 8keV - 18keV$
    - $Si\ 440 = 18keV - ???$
- Side effects:
  - GOOD : Increased Q range ( $Q_{min} \sim 2 \times 10^{-5} A^{-1}$ )
  - BAD : need for much better mechanical stability

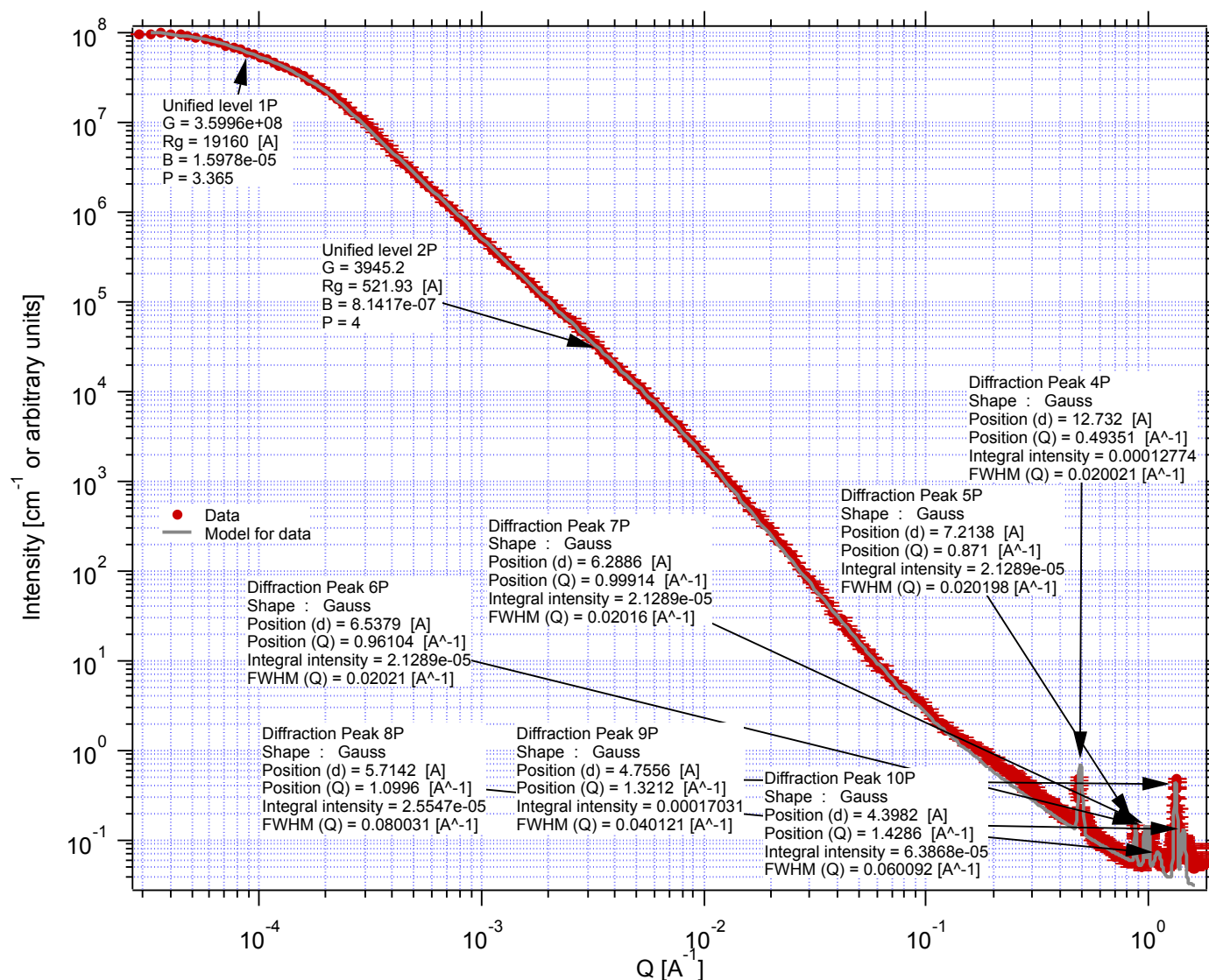
**Target : World-wide unique facility with capabilities matched by no one.**



Fri, Nov 4, 2011, 3:26:46 PM

# High-resolution / high-energy USAXS

Results in  
2012-1





## Current technical details

- intensity and Q range:
  - Up to **11 decades of intensity range**
  - **$1\text{e-}4 \text{ \AA}^{-1}$  to  $1 \text{ \AA}^{-1}$  Q range**  
 **$0.5 \text{ nm} \rightarrow >1 \text{ micron}$**
- Scan time : 10 min/scan  
(shortest scans down to 3 minutes)
- Flexible beam size  
 $1 \times 2 \text{ mm} \rightarrow 0.02 \times 0.2 \text{ mm}$
- Energy range : **10 – 18 keV**
- Q resolution ~  **$0.0001 \text{ \AA}^{-1}$**
- Geometries:
  - Slit smeared w/pinSAXS
  - 2D collimated
  - USAXS-XPCS for slow materials dynamics
  - USAXS-Imaging (imaging materials at various q vectors)

## High energy

- intensity and Q range:
  - Up to **11 decades of intensity range**
  - **$2\text{e-}5 \text{ \AA}^{-1}$  to  $2 \text{ \AA}^{-1}$  Q range**  
 **$0.3 \text{ nm} \rightarrow > 30 \text{ micron}$**
- Scan times as fast as 2 min/scan
- Flexible beam size  
 $1 \times 2 \text{ mm} \rightarrow 0.02 \times 0.2 \text{ mm}$
- Energy range : **10 – 30 keV**
- Q resolution ~  **$0.00002 \text{ \AA}^{-1}$**
- Geometries:
  - Slit smeared w/pinSAXS
  - 2D collimated
  - USAXS-XPCS for slow materials dynamics
  - USAXS-Imaging (imaging materials at various q vectors)

## Conclusions...

- USAXS is uniquely useful, with lot of users and documented impact in many, many areas of science

*“From Chocolate to Pu in one week”*

- USAXS is shown to be industrially robust, reliable instrument with high uptime (close to 100%) and user friendliness...
- Done a lot of unique work
- Generated publications (222 on my list 1999 – 2011)
- Enabled many student thesis
- Is available through GUP system to the best proposals...